

## **Total Sulfur (Sulfate and Elemental) in Dry Fertilizers**

### Scope

This method is only suitable for the determination of the amount of sulfate and elemental sulfur in dry fertilizers.

### Summary

The sample is digested with hydrochloric acid to dissolve sulfate sulfur which is separated from the residue by filtration and then precipitated as barium sulfate by the addition of barium chloride. Percent sulfate sulfur is based on the weight of barium sulfate. The residue on the filter contains any elemental sulfur. It is washed, dried, weighed and treated with CS<sub>2</sub> (carbon disulfide) to remove the elemental sulfur which is determined by difference in weight before and after treatment with CS<sub>2</sub>.

### Apparatus and Materials

- A. 500 ml erlenmeyer flask or 400 ml beaker.
- B. Gooch crucible.
- C. Glass fiber filter paper.
- D. 500 ml filter flask.
- E. Hot plate.
- F. Oven, 250°C.
- G. Whatman No. 42 filter paper.

### Reagents

- A. Concentrated hydrochloric acid.
- B. Barium chloride solution (10%): Dissolve 100 g of barium chloride dihydrate (BaCl<sub>2</sub>·2(H<sub>2</sub>O)) in 900 ml of deionized water. Filter through Whatman No. 42

filter paper.

#### Procedure

- A. Weigh a sample containing about 0.5 g of the ground sample into a 500 ml erlenmeyer flask or 400 ml beaker. Record the weight of the sample to the nearest tenth of a milligram.
- B. Add 200 ml of deionized water and 15 ml of concentrated hydrochloric acid to the flask.
- C. Heat the contents of the flask to boiling and boil gently for about 10 minutes.
- D. Filter the contents of the flask through a gooch crucible containing glass fiber filter paper and wash with hot deionized water. Set the washed crucible aside.
- E. Quantitatively transfer the filtrate to a 500 ml erlenmeyer flask or 400 ml beaker and bring to boiling.
- F. Add slowly, with constant stirring, a slight excess of 10%  $\text{BaCl}_2$  solution (about 15 ml).
- G. Digest the sample in the flask or beaker on a low temperature hot plate or a steam bath for one hour. **Important:** the temperature must be adjusted so that the solution does not boil.
- H. Let stand at room temperature overnight.
- I. Dry a gooch crucible and glass fiber filter paper for a minimum of 60 minutes at  $250^\circ\text{C}$ . Cool in a desiccator and weigh to the nearest tenth of a milligram.
- J. Filter the cooled solution and  $\text{BaSO}_4$  precipitate through the gooch and filter paper.
- K. Wash the precipitate, gooch and filter paper with 10 portions of hot deionized water.
- L. Dry the gooch, filter paper and precipitate at  $250^\circ\text{C}$  for a minimum of 60 minutes. Cool in a desiccator and weigh to the nearest tenth of a milligram.
- M. Wash the insoluble residue in the crucible (set aside in step D) with five 10 ml portions of acetone saturated with elemental sulfur.

- N. Dry 1 hour at 100°, cool in desiccator and weigh to the nearest tenth of a milligram.
- O. Wash the residue with three 5 ml portions carbon disulfide (CS<sub>2</sub>) and drain. **Note:** CS<sub>2</sub> is flammable and toxic so do the washing in a well-ventilated hood.
- P. Dry 1 hour at 100°, cool in desiccator and weigh to the nearest tenth of a milligram.
- Q. Perform a blank on the elemental portion of the method by weighing 5.0 g of a fertilizer sample containing no elemental sulfur and transferring to a 500 ml erlenmeyer or a 400 ml beaker.
- R. Treat this blank as in steps B-D above.
- S. Skip the sulfate sulfur steps (E-L) and proceed with steps M-P.

#### Calculations

- A. Calculate the % sulfate sulfur as follows:

$$\% \text{ Sulfate S} = \text{g BaSO}_4 \times 0.1374 \times 100 \div \text{g sample}$$

where:  $\text{g BaSO}_4 = \text{wt gooch} + \text{paper} + \text{precipitate (step L)} - \text{wt gooch} + \text{paper (step I)}$

- B. Calculate the elemental sulfur blank as follows:

$$\text{Elemental blank} = \text{wt gooch} + \text{residue before CS}_2 \text{ treatment (step N for blank run)} - \text{gooch} + \text{residue after CS}_2 \text{ treatment (step P for blank run)}$$

- C. Calculate the % elemental sulfur as follows:

$$\% \text{ Elemental sulfur} = \text{grams elemental sulfur} \times 100 \div \text{g sample}$$

where:  $\text{grams elemental sulfur} = \text{wt gooch} + \text{residue before CS}_2 \text{ treatment (step N for sample run)} - \text{gooch} + \text{residue after CS}_2 \text{ treatment (step P for sample run)} - \text{elemental blank (step B of calculations)}$

D. Calculate the % total sulfur as follows:

$$\% \text{ Total Sulfur} = \% \text{ Sulfate sulfur} + \% \text{ elemental sulfur}$$

#### Bibliography

Official Methods of Analysis (1984) 14th Ed., AOAC, Washington, D.C., sec. 2.183(a)